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Primary Gas Toxicities and Smoke Particle Characteristics During Combustion of Mine Ventilation Ducts

Development of a Test Parameter

By Maria I. De Rosa and Charles D. Litton

BUREAU OF MINES

UNITED STATES DEPARTMENT OF THE INTERIOR



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**UNITED STATES DEPARTMENT OF THE INTERIOR
Manuel Lujan, Jr., Secretary**

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T S Ary, Director**

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UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

°C	degree Celsius	L	liter
cm	centimeter	L/min	liter per minute
cm ² /p	square centimeter per particle	min	minute
cm ² /(p•g)	square centimeter per particle per gram	pct	percent
ft	foot	p/cm ²	particle per square centimeter
g	gram	ppm	part per million
in	inch	ppm/g	part per million per gram

PRIMARY GAS TOXICITIES AND SMOKE PARTICLE CHARACTERISTICS DURING COMBUSTION OF MINE VENTILATION DUCTS

Development of a Test Parameter

By Maria I. De Rosa¹ and Charles D. Litton²

ABSTRACT

The U. S. Bureau of Mines performed experiments to determine if the primary gas toxicities evolved during the early ($\sim 300^{\circ}\text{C}$) and later ($\geq 400^{\circ}\text{C}$) combustion stages of chlorinated polyester-reinforced mine ventilation ducts could be predicted by a smoke particle characteristic, for the development of a test parameter. The experiments were conducted in an approximately 20-L furnace, at 14-min durations at set furnace temperatures of 250°C and $1,000^{\circ}\text{C}$, with an airflow through the furnace of 10 L/min. The variables studied, as a function of time, were the hydrogen chloride (HCl), carbon monoxide (CO), carbon dioxide (CO_2), and oxygen (O_2) concentrations, furnace temperatures, the sample mass weight loss, the average smoke particle diameter (d_p) and average number and concentration (n_o), and the product of the average smoke particle diameter and concentration ($d_p n_o$). Results show that the inverse of the smoke particle diameter-concentration product ($1/d_p n_o$), correlates directly and significantly with the primary gas toxicities evolved during both early (HCl) and later (CO) stages of combustion.

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INTRODUCTION

In 1984, the U.S. Bureau of Mines, within its mission to create and maintain safety in the mines and, specifically, to assess the hazards of mine materials during fire, initiated a series of experiments to study smoke particle characteristics measured during the early ($\sim 300^\circ\text{C}$) combustion stage of mine combustibles. It was found (1)³ that smoke particles produced by different combustibles differed in average diameter (d_p), average number concentration (n_o), and the product of the smoke particle average diameter and concentration ($d_p n_o$).

A direct correlation was established (2) between the inverse of the smoke particle diameter-concentration product ($1/d_p n_o$), measured during the combustion of various materials, and the toxicity data obtained with similar materials and reported by Anderson and Alarie (3) (animal study) and Paciorek (4) (gas analyses). In subsequent studies (5-8), simultaneous measurements were

made of primary gas concentrations and smoke particle characteristics during the combustion of polyvinyl chloride (PVC) and other chlorinated mine conveyor belts, PVC and other chlorinated mine brattices, wood, and coal; it was found that the inverse of the smoke particle diameter-concentration product ($1/d_p n_o$), correlated directly and significantly with those toxicities.

In this study, the Bureau set out to correlate the smoke particle characteristic $1/d_p n_o$ with the primary gas toxicities evolved during the early ($\sim 300^\circ\text{C}$) and later ($\geq 400^\circ\text{C}$) combustion stages of chlorinated polyester, fiberglass-reinforced ventilation duct samples. These correlations would allow the development of a test parameter for assessing simply, and inexpensively, the toxic hazard of ducts during stages of fire when miners must plan and prepare for escape.

BACKGROUND

Organic materials when heated release gaseous species whose toxicity and rates of release depend on the nature of the material, on temperature, and on material ease of thermal decomposition (5). Synthetic polymeric materials, because of resin composition, release toxic gases earlier, and at faster rates, because the cohesive and covalent bonds of the polymer chain break easily under heat penetration (9). Polyester-reinforced mine ventilation ducts are made of polyester resin, derived from the reaction of a dibasic acid (maleic or phthalic anhydride) with a dibasic dihydric alcohol (propylene or ethylene glycol), blended with a reactive vinyl monomer (styrene, vinyl benzene, or vinyl toluene), and reinforced (>80 pct) with fiberglass, treated with inhibitors (phenolic compounds) and flame retardants (chlorine, phosphorous, and antimony trioxide). However, even the finished products can be ignited by sufficient heat sources (10), releasing at lower temperatures hydrogen chloride (HCl), and at higher temperatures, carbon monoxide (CO). Table 1 reports the toxicity of pyrolysis products of polyester fibers (11). HCl, with a short-term exposure limit (STEL) of 25 ppm and immediately dangerous to life and health (IDLH) at 100 ppm as hydrochloric acid, deposits on soot particles, which are transported to the mucous membranes, causing irritation and severe damage to the eyes and to the lower respiratory tract, with ensuing cough, dizziness, heaviness of the legs, impaired vision, and disorientation (12). The pulmonary damage from polymers containing HCl has been shown (13-14) to be more severe than that from HCl alone, probably because the particulates may be drawn into and deposited into the lungs, where the absorbed and/or

dissolved HCl enhances the toxic effects. The HCl LC50 (lethal concentration at which 50 pct of laboratory animals die) has been found (15) to decrease from 5,600 ppm for pure HCl gas to 1,900 ppm for PVC products, and the HCl LC50 for cannulated animals (simulating human breathing habits) has been found (16) to be 1,000 ppm as opposed to 10,000 ppm for noncannulated animals; humans tend to breathe through the mouth in the presence of such irritants. Furthermore, the HCl may also be condensed by moist air to form a corrosive hydrochloric acid mist that, together with smoke, can obscure vision, interfering with escape and rescue.

At higher temperatures ($\geq 400^\circ$), which during fire may follow in a matter of minutes (10), the dehydrochlorinated residue yields large quantities of carbon monoxide (CO) (STEL = 400 ppm; IDLH = 1,500 ppm). CO, easily absorbed through the lungs into the blood to form carboxyhemoglobin (COHb), blocks the oxygen carriage to the brain, causing disorientation, vertigo, and cardiovascular and neurological complications (17). The COHb content of laboratory animals dead in PVC fires was found to be 13 to 30 pct compared with the expected 85 pct for pure CO (18). The CO LC50 in PVC fires was found to be 900 ppm compared with 4,600 ppm for pure CO (19).

Table 1.—Toxicity of pyrolysis products of 10-g polyester fibers (11)

Pyrolysis temp	$^\circ\text{C}$. .	500
Maximum CO	ppm . .	2,900-5,300
COHb ¹	pct . .	20-30
Minimum O ₂	pct . .	15.6-19.7
Time to incapacitation	min . .	15-19

¹Carboxyhemoglobin of 1 male rat placed in a 13-L exposure chamber connected by a hose to a 45-L combustion chamber.

³Italic numbers in parentheses refer to items in the list of references at the end of this report.

EXPERIMENTAL SYSTEM

The system (fig. 1) consists of an approximately 20-L furnace whose temperature during experiments rises automatically from ambient at a rate depending on the set temperature, which ranges from 100° to 1,200° C (fig. 2). A universal load cell, located under the furnace floor, contacts a sample cup, which transmits sample weight loss (voltages) via a bridge amplifier to a strip chart recorder. A vacuum pump draws ambient air (10 L/min) continuously into the furnace, via an opening at the center of the furnace door, and pumps combustion air into the CO,

CO₂, and O₂ gas analyzers, and into the submicrometer particle detector analyzer (SPDA) (1.6 L/min) (20). The SPDA voltages are used to compute the average smoke particle diameter (d_g), the average number and concentration (n_o), and the product of the average smoke particle diameter and concentration ($d_g n_o$). A data acquisition system continuously acquires, computes, and stores data of sample and furnace temperatures, airflows, gas concentrations, weight losses, and the SPDA initial and experimental voltages. The stored data are then plotted.

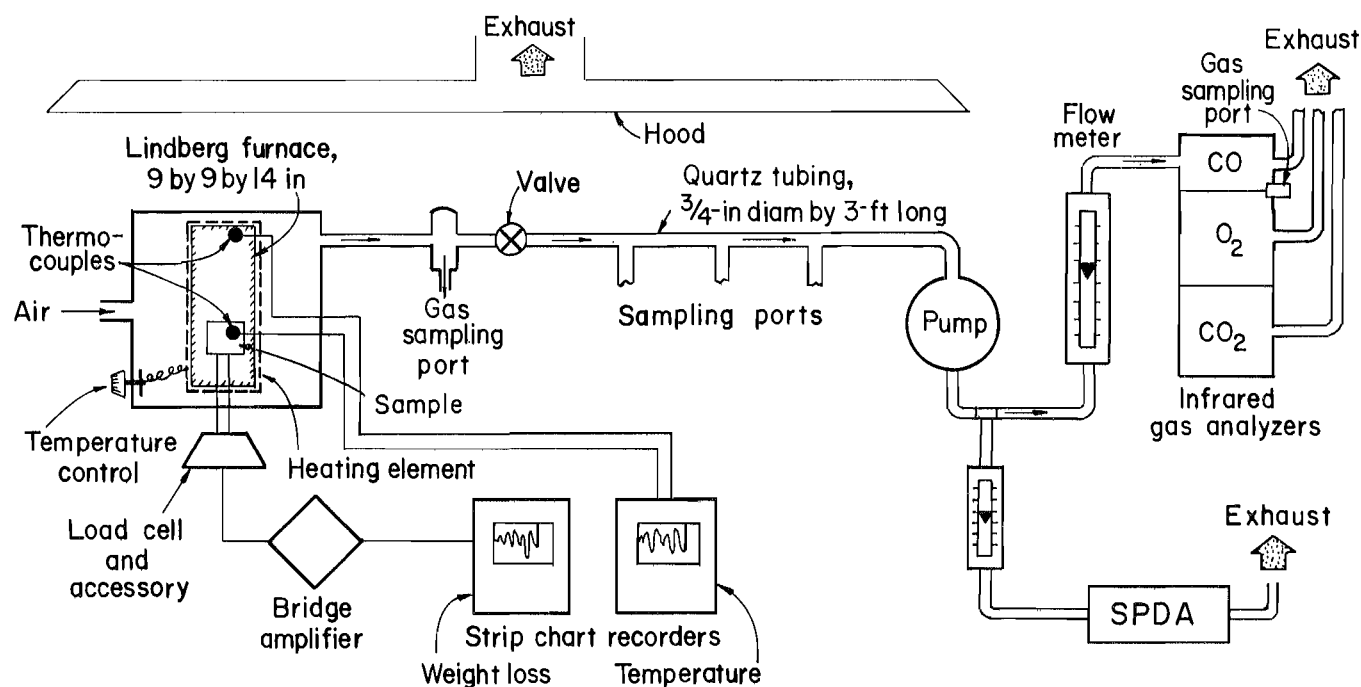


Figure 1.—Experimental system.

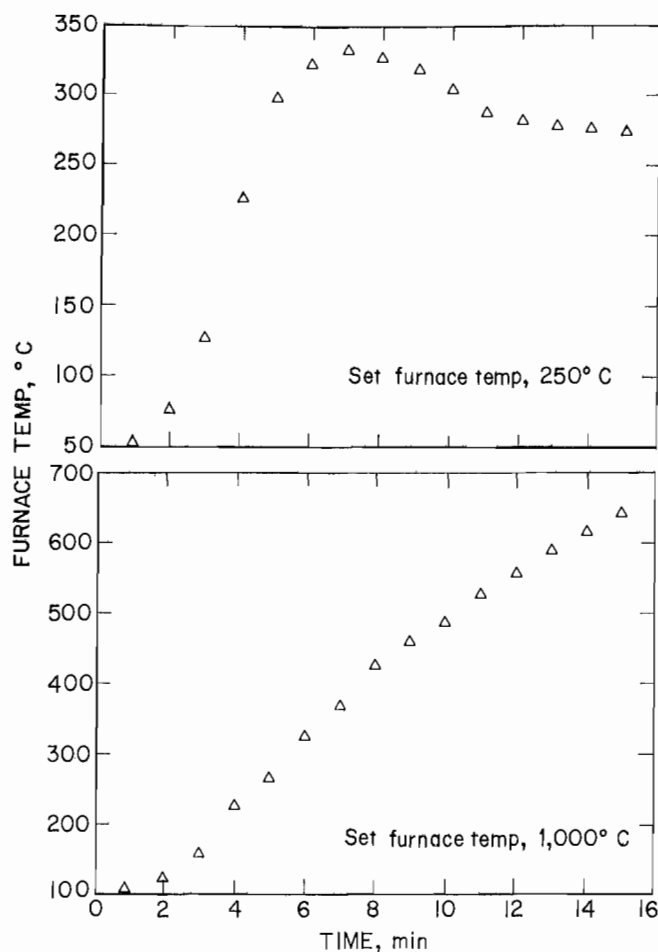


Figure 2.-Furnace temperature versus time at set furnace temperatures of 250° and 1,000° C.

DATA REDUCTION AND ANALYSES

HCl and CO concentrations were measured at 250° C set furnace temperature and 14-min test duration (representing an early stage of combustion, at which most of the HCl evolves with no gross degradation of the sample). The HCl and CO concentrations observed at 0.1 g of sample weight loss, approximately at the 10th minute, adjusted to 1 g of weight loss, yielded the HCl and CO load values (in parts per million per gram). The load value, derived at a specific time and airflow, is important toxicological parameter, for it establishes a priori the total toxic gas concentrations expected to evolve at each stage of combustion for each material: HCl evolving during the early stage (~300° C) of combustion, because of an early and fast dehydrochlorination process (CO concentrations are insignificant at this stage); CO evolving at a later stage ($\geq 400^\circ$ C) of combustion, because of gross degradation of the sample. Therefore, the HCl load value is predictive of the toxic hazard that the polyester chlorinated ducts pose during the early stage of combustion; the combined HCl and CO load values are

predictive of the total toxic hazard that the ducts pose during the later stages of combustion. At 250° C conditions, the average smoke particle diameter (d_g), average number and concentration (n_o), and the product of the average smoke particle diameter and concentration ($d_g n_o$) were measured. The $d_g n_o$ is obtained from the ratio of the SPDA experimental and initial current output (I_e/I_o) following the relationship in equation 1. See also figure 3.

$$I_e/I_o = 1/(K d_g n_o) (1 - \exp(-K d_g n_o)), \quad (1)$$

where K = charging constant, $0.012 \text{ cm}^2/\text{p}$.

The total $d_g n_o$ values observed during each experiment for each material, adjusted to 1 g of sample mass weight loss, yielded the $d_g n_o$ load value (in particles per square centimeter per gram). Its inverse, $1/d_g n_o$ load (in square centimeters per particle per gram), has been correlated with the HCl load and with the combined HCl and CO

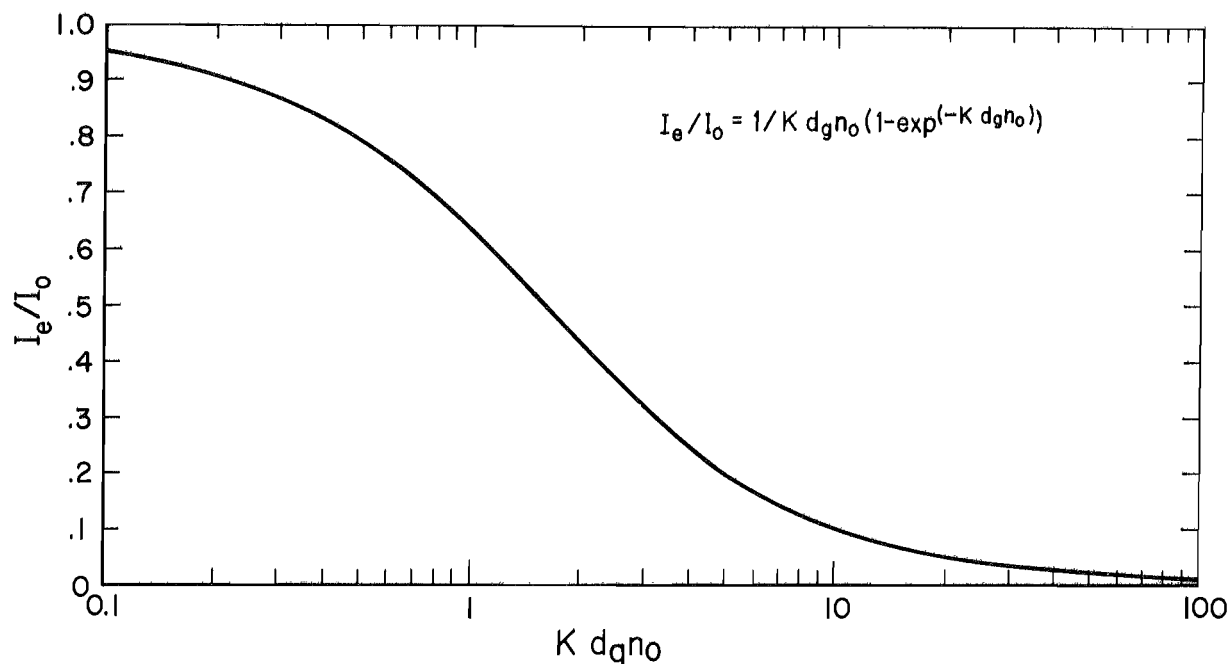


Figure 3.—SPDA current ratio as function of smoke particle diameter-concentration product.

combined HCl and CO loads (the CO load values having been previously normalized to HCl values by scaling them down by a factor of 15, because CO is at least 15 times less toxic than HCl). The $1/d_g n_o$ load values have been also correlated with the combined HCl and CO load values scaled to their STEL and IDLH values.

At 1,000° C set furnace temperature and 14-min test duration (representing the later stage of combustion at which most of the CO evolves, because of gross degradation of the sample) CO and other gas concentrations, sample weight loss, and furnace temperatures were measured.

EXPERIMENTAL PROCEDURE

Two sets of experiments (five experiments in each set, each experiment repeated three times) were performed at set furnace temperatures of 250° C (maximum furnace temperature, 300° C at the 7th minute) and 1,000° C (maximum furnace temperature, 600° C at the 14th minute) for a 14-min duration, with samples of polyester-reinforced mine ventilation ducts. A list of the materials is reported in table 2. A 1-g sample was placed in a 2.5-cm-diam cup, the furnace was set at the desired temperature, and the furnace (10 L/min) and the SPDA (1.6 L/min) airflows were also set. Syringe grab samples, stored in vacuum containers, were analyzed for CO, CO₂, and O₂ by gas chromatography; HCl concentrations were determined by short-term exposure tubes. The chlorine (Cl₂) content of the material was determined by the bomb-combustion, selective-electrode method; the antimony trioxide (Sb₂O₃) content of the material was obtained from the manufacturer. Other variables measured, as a function

of time, were the sample and furnace temperatures, sample mass weight loss, and the SPDA initial voltages (without smoke) and experimental voltages (with smoke).

At 1,000° C set furnace temperature and 14-min duration, CO, CO₂, and O₂ concentrations (by syringe grab samples), and sample mass loss were measured.

Table 2.—Materials Investigated

Polyester-reinforced ventilation duct ¹	Cl ₂ , pct
D1	10.5
D2	4.7
D3	4.3
D4	3.8
D5	4.7

¹Polymer component is polyester resin reinforced (>80 pct) with fiberglass fibers, treated for flame retardancy with chlorinated additives and antimony trioxide. All materials contained 5 pct Sb₂O₃.

RESULTS AND DISCUSSION

At 250° C set furnace temperature, HCl was found to be the primary gas released for all ducts, depending on the percentage of chlorinated additives of the material; the time and rates of release depended on the ease of thermal material decomposition. At this stage of combustion, small CO concentrations were released by all ducts because of low thermal decomposition of the samples. The higher the HCl values, the higher the $1/d_g n_o$ values.

At 1,000° C, large CO concentrations were released by all duct samples because of complete and fast combustion of the samples (only fibers remained).

Correlations (correlation coefficient (r) = 0.85) were established between the $1/d_g n_o$ load and the HCl load (fig. 4), between the $1/d_g n_o$ load and the combined HCl and CO loads (fig. 5), between the $1/d_g n_o$ load, the combined loads, and the HCl load (fig. 6), and between the $1/d_g n_o$ load and the combined loads scaled to their STEL and IDLH values (fig. 7).

Figure 4 shows that sample duct D1, with a chlorine content of approximately 10 pct, yielded, by far, the highest HCl load values and the highest $1/d_g n_o$ load values (see

also table 3); also, duct D1 yielded the highest combined CO and HCl load values (fig. 5). Figure 6 shows that the CO load values, normalized to HCl values, did not significantly alter the HCl load values for all samples. Finally, as expected, duct D1 contained by far the highest number of STEL and IDLH values (fig. 7). Table 4 shows total sample weight losses and CO concentrations. At 250° C set furnace temperature, all ducts underwent little (> 15 pct) thermal decomposition, accompanied by low CO concentrations. Maximum CO concentrations of approximately 60 ppm were released at the 10th minute by ducts D1 and D2.

At 1,000° C set furnace temperature, all samples underwent total thermal decomposition; the percentage of sample mass not decomposed contained only glass fibers, yielding large CO concentrations. The largest CO concentrations (approximately 1,500 ppm), however, were released at the 10th minute by ducts D1 and D3. Figure 8 shows smoke particle diameter-concentration product ($d_g n_o$) versus time at 250° C. Figure 9 shows CO concentrations versus time at 250° and 1,000° C.

Table 3.—Oxidative thermal degradation data at 250° C with furnace airflow of 10 L/min

Polyester-reinforced ventilation duct ¹	HCl, ppm	CO, ppm	WL, g	Time, min	Load					
					² HCl, ppm/g	² CO, ppm/g	³ $1/d_g n_o$, cm ² /(p·g)	⁴ HCl and CO, ppm/g	⁵ HCl and CO	
									STEL	IDLH
D1, 10.5 pct Cl ₂	40	130	0.1	10	400	1,300	0.0009	487	19.5	5
D2, 4.7 pct Cl ₂	15	84	.1	10	150	840	.0003	165	7	2
D3, 4.3 pct Cl ₂	11	120	.1	10	110	1,200	.00025	123	5	1.2
D4, 3.8 pct Cl ₂	10	88	.1	10	100	880	.0002	119	5	1.2
D5, 4.7 pct Cl ₂	15	83	.1	9	150	830	.0003	165	7	1.7

IDLH Immediately dangerous to life and health.

STEL Short-term exposure limit.

WL Weight loss.

¹1-g sample.

²Each load value derives from the concentration observed at 0.1 g of sample weight loss per gram of weight loss, at a specific time and dilution air.

³Inverse of $d_g n_o$ load. Each $d_g n_o$ load is derived from total $d_g n_o$ values per gram of sample weight loss.

⁴Each value derives from the combined HCl and CO load values. Each CO load value has been previously normalized to HCl values by scaling it down by a factor of 15, because CO is at least 15 times less toxic than HCl.

⁵STEL and IDLH values contained in each combined load (STEL, 25 ppm ; IDLH, 100 ppm).

Table 4.—Comparison of oxidative thermal degradation data at 250° and 1000° C, with furnace airflow of 10 L/min

Polyester-reinforced ventilation duct ¹	250° C						1,000° C					
	CO, ppm	Time, min	O ₂ , pct	CO ₂ , ppm	Total WL, g	Furnace temp, °C	CO, ppm	Time, min	O ₂ , pct	CO ₂ , ppm	Total WL, g	Furnace temp, °C
D1	6	6	21	500	0.13	266	10	6	20.8	700	0.41	280
	9	8	20.8	500		320	50	8	20.7	710		380
	25	9	20.8	550		318	600	9	20.7	990		430
	60	10	20.8	550		311	1,100	10	20.7	1,200		450
	18	12	21	520		295	520	12	20.7	1,100		530
	12	14	20.8	500		280	320	14	20.7	1,100		600
D2	6	6	21	500	.12	273	10	6	20.8	690	.33	270
	8	8	21	520		323	49	8	20.8	670		370
	33	9	21	550		318	616	9	20.7	960		420
	64	10	21	520		311	948	10	20.7	1,190		460
	14	12	20.8	500		295	417	12	20.8	1,010		535
	12	14	21	500		281	303	14	20.7	1,020		600
D3	7	6	21	520	.11	269	5	6	21	560	.41	273
	8	8	20.8	550		323	219	8	20.8	750		373
	7	9	20.8	580		320	646	9	20.7	920		417
	20	10	21	600		313	1,340	10	20.7	1,200		459
	14	12	20.8	580		298	577	12	20.7	930		534
	8	14	20.8	530		285	343	14	20.7	1,360		599
D4	4	6	21	660	.14	266	8	6	20.8	620	.64	275
	10	8	21	660		321	48	8	20.8	700		376
	13	9	21	640		318	369	9	20.8	910		420
	28	10	21	640		311	764	10	20.7	1,130		460
	20	12	21	600		296	478	12	20.7	6,150		540
	12	14	21	600		281	369	14	20.7	1,980		605
D5	5	6	21	550	.10	260	19	6	20.9	570	.41	265
	18	8	21	550		323	500	8	20.8	600		263
	23	9	21	580		319	739	9	20.7	1,160		406
	13	10	21	560		312	837	10	20.5	2,500		447
	7	12	21	600		297	503	12	20.6	1,770		526
	5	14	21	530		282	510	14	20.6	1,670		558

WL Weight loss.

¹1-g sample.

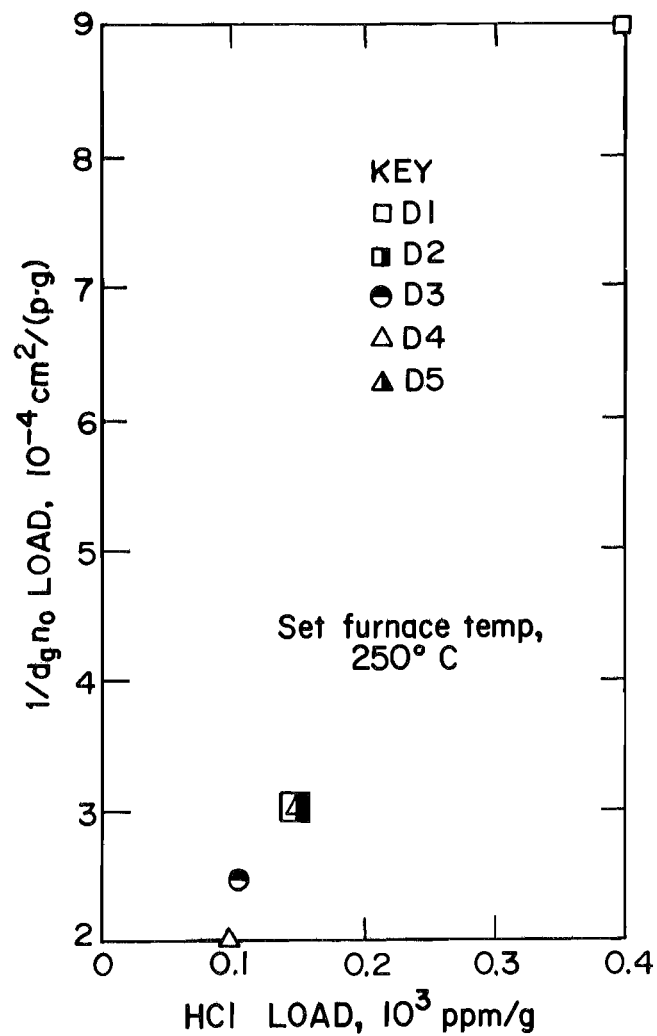


Figure 4.-Inverse of smoke particle diameter-concentration product ($1/d_g n_o$) load versus HCl load at 250° C.

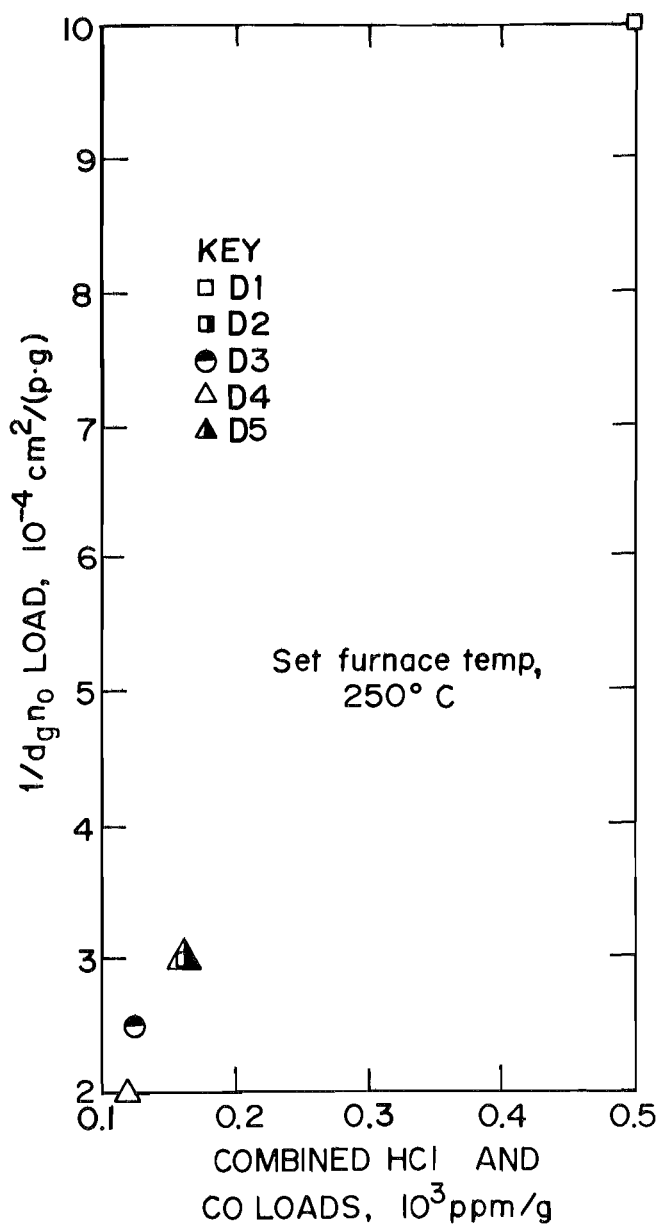


Figure 5.-Inverse of smoke particle diameter-concentration product ($1/d_g n_o$) load versus combined HCl and CO loads at 250° C.

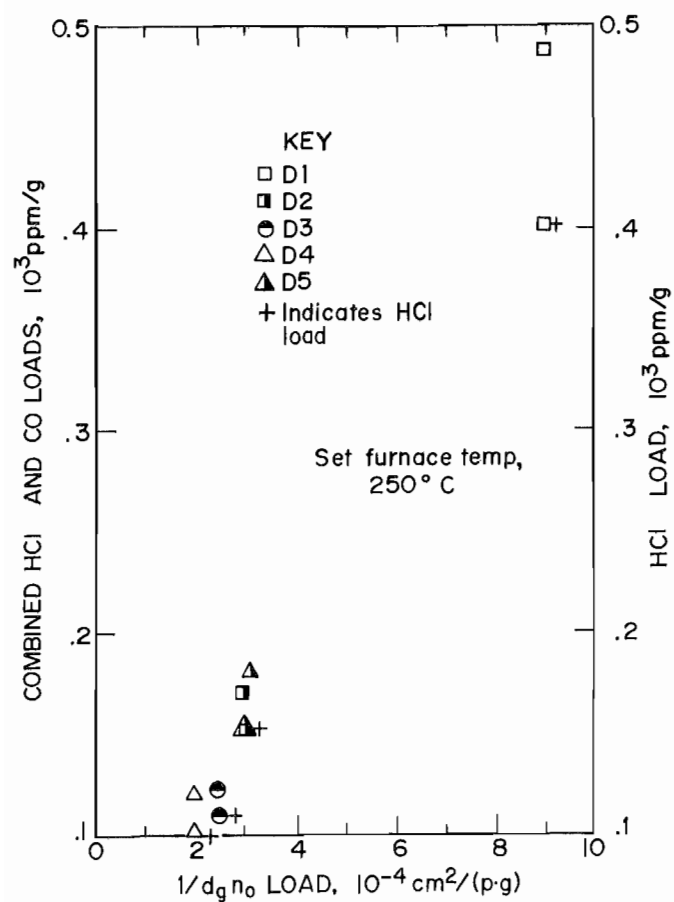


Figure 6.—Inverse of smoke particle diameter-concentration product ($1/d_g n_o$) load versus combined loads and versus HCl load at 250°C.

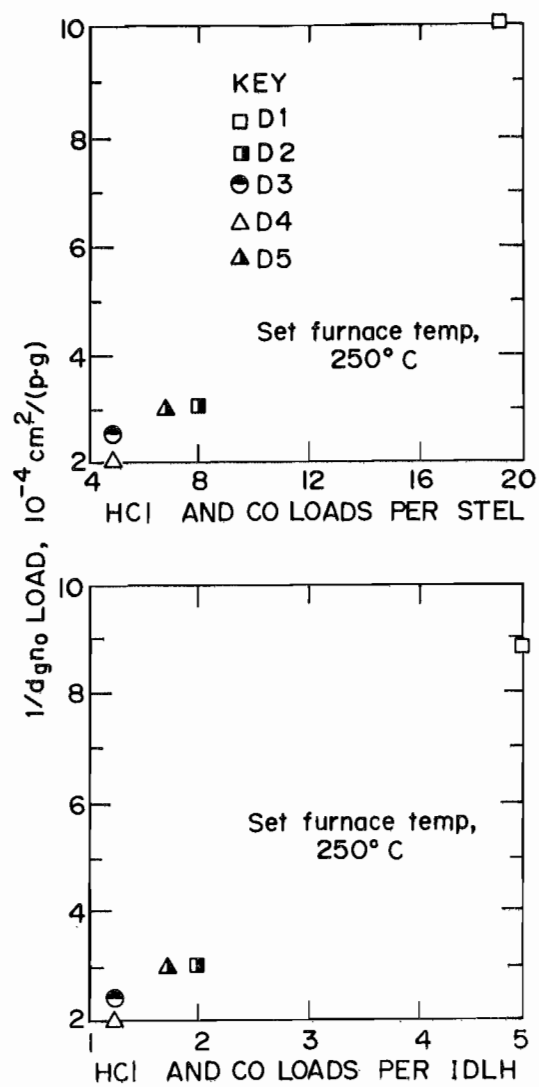


Figure 7.—Inverse of smoke particle diameter-concentration product ($1/d_g n_o$) load versus combined HCl and CO loads scaled to STEL and IDLH values at 250°C.

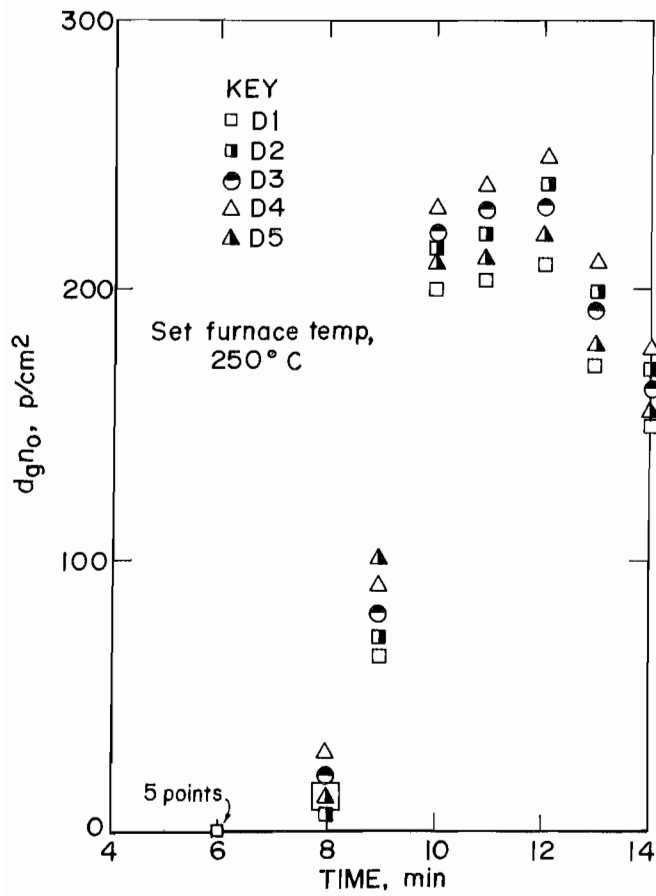


Figure 8.—Smoke particle diameter-concentration product ($d_g n_o$) versus time at 250°C.

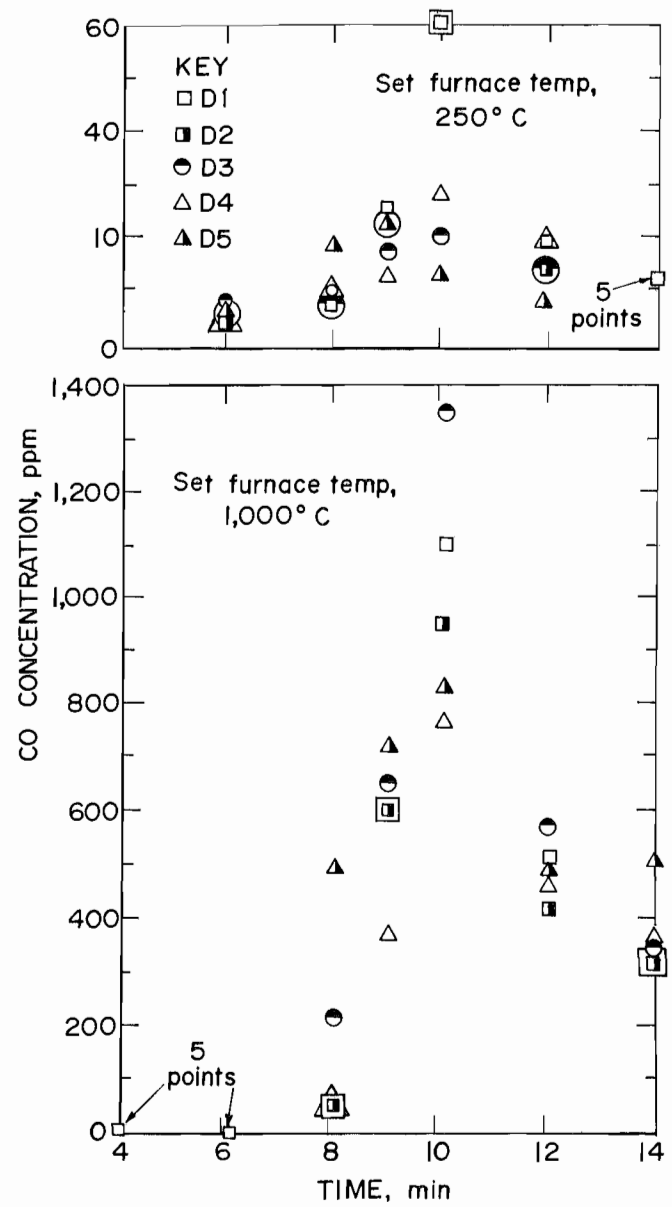


Figure 9.—CO concentrations versus time at 250° and 1,000° C.

CONCLUSIONS

According to the Bureau's findings, the inverse of the smoke particle diameter-concentration product ($1/d_p n_o$) load correlates directly and significantly with the HCl and CO toxicities; HCl being the primary toxicity evolving during the early ($\sim 300^\circ\text{C}$) stage of combustion depending on the percentage of chlorinated additives of the material (CO concentrations are minimal at this stage), and CO being the primary toxicity evolving in large quantities

during a later ($\geq 400^\circ\text{C}$) stage of combustion, which during fire can develop in a matter of minutes.

The excellent correlation of the inverse of the smoke particle diameter-concentration product ($1/d_p n_o$) with the primary gas toxicities suggests its use as a test parameter to assess simply and inexpensively the toxic hazard that ducts pose during fire.

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